ANALYSIS OF THE PRECIPITATION REACTOR-SEPARATION EQUIPMENT SYSTEM. CONTINUOUS REACTOR AND THE ROTARY VACUUM FILTER OPERATING AT THE SELECTED NEGATIVE PRESSURE DROP

Otakar Söhnel

Research Institute of Inorganic Chemistry,
Chemopetrol, 400 60 Ústi nad Labem

An analysis has been performed of the continuous precipitation reactor — rotary vacuum filter system (operating at the selected negative pressure drop) on the basis of the unit output. Filtration area necessary for separation of the product from the precipitation reactor is a function of the mean residence time of suspension in the reactor, concentration of the precipitating solutions, porosity of the filtration cake and the filtration negative pressure drop. Application of the derived relations is demonstrated on the continuous precipitation of $Mg(OH)_2$.

Design of industrial crystallisers is rather well worked out at present[1]. The situation is usually simplified due to the fact that the crystallisation product is relatively coarse and its separation from the mother liquor presents no problems for usual types of filtration centrifuges. In this case the main attention may be focused to the technology of crystallisation neglecting the succeeding separation. Therefore the attempts on optimisation of the crystalliser-filter system have not led to any conclusions[2].

Quite different situation is met at precipitation of sparingly soluble compounds from highly supersaturated solutions. The formed particles of the solid phase are usually rather small due to which the separation is complicated and even becomes the limiting factor in the technology of precipitation. Therefore, from the chemical engineering point of view, the system consisting of precipitation reactor and separation unit should be regarded as a single process unit. Its operating conditions should be determined in such a way that the output of the unit becomes optimal. No procedure however, has been proposed which would enable determination of both the best precipitation conditions from the view point of the unit output and the well balanced dimensions of individual equipments consisting a unit.

This paper presents analysis of the continuous precipitation reactor-rotary drum vacuum filter system as concerns the system output and determination of suitable dimensions of individual units which are mutually balanced as concerns their output.

THEORETICAL

The mass specific filtration resistance α of the suspension depends on properties of the suspended solid phase according to the Carman–Kozeny equation[3]

$$\alpha = 5(1 - \varepsilon)\, s^2/\varrho\varepsilon^3 . \tag{1}$$

Specific surface area of the solid phase produced by continuous precipitation, at the assumption of crystal growth controlled by the diffusion mechanism, is given[4]

$$s = (4k_a/3k_v \sqrt{\pi})\, [2Dv_{mol}(c - c_{eq})\, t_z]^{-1/2} . \tag{2}$$

Combination of Eqs (1) and (2) yields and expression relating the filtration resistance and conditions prevailing during continuous precipitation, i.e.

$$\alpha = [1{\cdot}415k_a^2(1 - \varepsilon)/k_v^2 \, . \, \varepsilon^3 Dv_{mol}\varrho(c - c_{eq})]\, . \, t_z^{-1} \tag{3}$$

or

$$\alpha = A \, . \, t_z^{-1} , \tag{4}$$

where A defined by the term in brackets of Eq. (3) is a function of the intial concentration of precipitated solutions through ε and $(c - c_{eq})$. The relation experimentally obtained for $Mg(OH)_2$ can be written[4]

$$\alpha = A \, . \, t_z^n , \tag{5}$$

where $n \neq -1$ due to compressibility of the filtration cake. The majority of filtration cakes is compressible and thus a general validity of Eq. (5) can be expected which will be in the following text used instead of theoretical Eq. (4).

Specific output of the rotary vacuum filter is expressed by relation

$$q_f = \frac{G}{t_f} = 2 \, \{\Delta Px_s\varrho(1 - \varepsilon)/\eta\alpha\, t_f[\varrho(1 - \varepsilon) - x_s]\}^{1/2} . \tag{6}$$

As quantities α, ε, η and x_s (if thickening of suspension before filtration is not considered) are functions of precipitation conditions, q_f depends also on conditions prevailing during precipitation. Relations between the initial concentrations of solutions precipitated in stoichiometric ratio and individual quantities are as follows

$$x_s = c_0 M_w/2 \tag{7}$$

$$\varepsilon = a + bc_0 \tag{8}$$

$$\alpha = A(c_0) \cdot t_z^{n(c_0)} \tag{9}$$

$$\eta = f(c_0), \tag{10}$$

where a, b, $A(c_0)$ and $n(c_0)$ must be determined for each compound experimentally while viscosity of the filtrate is mostly available in the reference literature.

Specific output of the reactor, q_r, is given by relation

$$q_r = P/V_r = x_s v/V_r = x_s/t_z . \tag{11}$$

The studied system is balanced as concerns the output if

$$q_r V_r = q_f A_f . \tag{12}$$

The quantity significant for design of the discussed system is

$$A_f/V_r = q_r/q_f = (t_f/2 \, \Delta P)^{1/2} \{\eta A x_s [a' + x_s(b' - 1)] /(a' + b'x_s)\}^{1/z} ,$$
$$\cdot t_z^{(n/2 - 1)} \tag{13}$$

where

$$\varrho(1 - \varepsilon) = a' + b'x_s \tag{14}$$

is obtained from Eqs (7) and (8).

The total filtration area necessary for processing of the production P is then

$$A_f = (q_r/q_f) Pt_z/x_s . \tag{15}$$

TABLE I

Parameters of Eq. (13) for continuously precipitated $Mg(OH)_2$[5,6]

c	x_s	A	n	η
0·5	14·58	$1·473 \cdot 10^{15}$	−1·5589	$9·3 \cdot 10^{-4}$
0·75	21·87	$2·007 \cdot 10^{16}$	−1·5713	$9·5 \cdot 10^{-4}$
1·0	29·16	$1·734 \cdot 10^{16}$	−1·7277	$9·71 \cdot 10^{-4}$
1·5	43·76	$1·734 \cdot 10^{16}$	−1·7277	$10·21 \cdot 10^{-4}$

RESULTS AND DISCUSSION

Application of the derived relations is demonstrated on the example of continuous precipitation of $Mg(OH)_2$ (ref.[4,5]). Precipitation has been performed in the continuous stirred reactor at 25°C fed simultaneously with $MgCl_2$ solution of the concentration c_0 and the same volume of NaOH solution with the concentration $2c_0$. The reaction took place at stoichiometric conditions. The results of experimental studies of $Mg(OH)_2$ precipitation given in Table I are valid only for the used filtration negative pressure drop $\Delta P = 30$ kPa and temperature 25°C as both α and ε are functions of negative pressure drop and temperature. Viscosity of the filtrate, *i.e.* of the NaCl solution with concentration $2c_0$ has been taken from literature[6]. Relation (8) for $Mg(OH)_2$ at the given conditions is[5]

$$\varepsilon = -0.063c_0 + 0.927 \quad 0.3 \leqq c_0 \leqq 1.5 \tag{16}$$

so that for $\varrho = 1\,930$ kg m^{-3} the constants of Eq. (14) are equal to $a' = 141.488$ and $b' = 4.169$.

As both $A(c_0)$ and $n(c_0)$ have been determined only for some selected values of c_0 and have not been a single valued (unique) function of c_0 the following calculations have been performed only for the actually measured concentrations of solutions.

The ratios (A_f/V_r) calculated from Eq. (13) for different conditions of precipitation and $t_f = 240$ s are given in Table II. This ratio is the filtration area in m^2 maximally required for separation of the production from one m^3 of the precipitation reactor. Total filtration area required for separation of production $P = 1$ kg s^{-1} of dry $Mg(OH)_2$ from a reactor has been calculated according to Eq. (15). Dependence of the maximum needed filtration area on studied precipitation parameters *i.e.* initial concentration of solution and the mean residence time of suspension in the reactor, is plotted in Fig. 1 where also the volume of the reactor necessary for production of $P = 1$ kg s^{-1} at the given precipitation conditions is shown.

TABLE II

Values of $(A_f/V_r)^a$ calculated from Eq. (13) for $\Delta P = 30$ kPa and $t_f = 240$ s

t_z	$c = 0.5$	$c = 0.75$	$c = 1.0$	$c = 1.5$
100	75.18	329.88	247.37	306.47
500	4.29	18.63	12.32	15.26
1 000	1.25	5.40	3.38	4.19
2 000	0.36	1.57	0.93	1.15

^{a} Dimension in m.

The initial concentrations of solutions in Fig. 1 have been expressed as x_s by use of Eq. (7); concentrations $c_0 = 0.5$; 0.75; 1.0 and 1.5 mol l^{-1} correspond to $x_s = 14.58$; 21.87; 29.16 and 43.76 kg m^{-3} of suspension. The calculated filtration area is the maximum value because the longest possible filtration time for the rotary filter $(t_f = 240$ s$)$ has been chosen. As the minimum rotation speed of the rotary filter drum is $r = 5$ rev. h^{-1} and filtration always takes place only on about $1/3$ of the filtration area, the maximum time available for filtration is

$$t_f = 3\,600\psi/r = 240 \text{ s}. \tag{17}$$

By increasing the speed of rotation of the drum t_f decreases and according to Eq. (6) q_f increases whereas filtration area A_f necessary for separation of production P decreases.

From Fig. 1 is obvious that while the volume of the reactor needed for production P decreases with decreasing t_z and increasing x_s, the needed filtration area for handling the production is passing through a maximum at precipitation of solutions with the concentration $c_0 = 0.75$ mol l^{-1} *i.e.* for $x_s = 21.87$ kg m^{-3} of suspension. For de-

FIG. 1
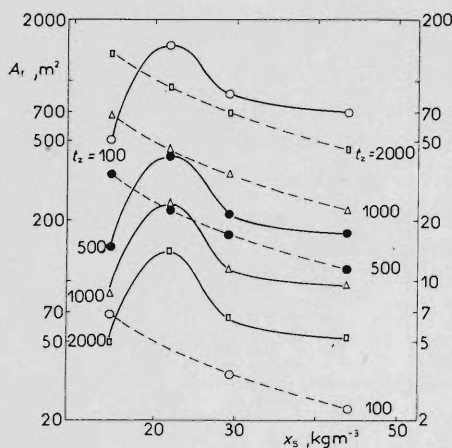
Maximum filtration area A_f (———) and volume of reactor V_r (— — —) necessary for production of 1 kg s^{-1} of dry Mg(OH)$_2$ as function of initial concentration of precipitated solutions (x_s) for different mean residence times of suspension in reactor (t_z)
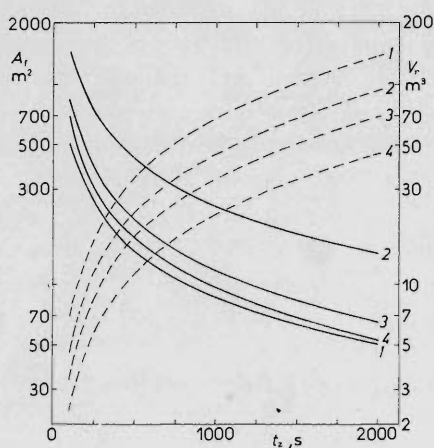
FIG. 2

Maximum filtration area A_f (———) and volume of reactor V_r (— — —) for production of 1 kg s^{-1} of dry Mg(OH)$_2$ as function of mean residence time of suspension in reactor for various initial concentrations of solutions. x_s: Curve *1* 14.58; *2* 21.87; 29.16, 43.76

sign of the precipitation reactor-separation unit system also the size of the reactor must be taken into consideration, which at low concentrations of precipitated solutions increases into unrealistic values. Interdependence of A_f and V_r is illustrated by Fig. 2, showing these quantities as function of the mean residence time of suspension in the reactor for various initial concentrations or precipitated solutions. The best combination of V_r and A_f for obtaining the production P then results from Fig. 2 if supplementary criteria, such as economic parameters, free area available for construction, equipments which are available *etc.* are introduced.

A critical t_z, dependent on concentration of precipitated solutions exists at continuous precipitation[5,7,8]. For $t_z > t_{z,\text{crit}}$ the α of the originating suspension is not decreasing any more and Eq. (5) therefore loses its validity. Exceeding the critical t_z results in only "empty" increase of V_r without any favourable effect on decrease of A_f.

It is necessary to stress that the indicated experimental expressions for $Mg(OH)_2$ are valid only for the actually used negative pressure drop $\Delta P = 30\,\text{kPa}$. But as α and ε are functions of ΔP, the actually used ΔP need not neccessarilly present the optimal value for filtration ensuring the smallest filtration area. The complete optimisation of the system can be performed only when variable ΔP is considered. Neither other precipitation conditions could be neglected, such as temperature, non-stoichiometric precipitaticn *etc.* which could significantly affect α of the originating suspension[4,9]. Selection of the optimum of precipitation technology resulting in production of the required modification of the compound in the process unit of the smallest possible size is thus a complex problem whose solution, however, is possible on basis of laboratory scale experiments.

LIST OF SYMBOLS

a, a'	constant in Eqs (8) and (14)
A	constant in Eq. (5) (m s kg^{-1})
A_f	filtration area (m^2)
b, b'	constant in Eqs (8) and (14)
c	concentration (mol m^{-3})
c_0	inlet concentration of precipitated solutions (mol m^{-3})
c_{eq}	equilibrium concentration (mol m^{-3})
D	diffusion $(\text{m}^2\,\text{s}^{-1})$
G	mass of dry material per 1 m^2 filter area (kg m^{-2})
k_a, k_v	surface and volume shape factors
M_w	molecular mass (kg mol^{-1})
n	exponent in Eq. (5)
ΔP	filtration negative pressure drop (vacuum) (k Pa)
P	production rate (kg s^{-1})
q_f	specific output of the filter $(\text{kg m}^{-2}\,\text{s}^{-1})$
q_r	specific output of the reactor $(\text{kg m}^{-3}\,\text{s}^{-1})$
r	number of rotations per h (h^{-1})

s specific surface area (m^{-1})

t_z mean residence time in the reactor (s)

t_f time of filtration (s)

v volumetric feed rate of liquid into reactor $(m^3 s^{-1})$

v_{mol} molar volume of solid $(m^3 mol^{-1})$

V_r volume of reactor (m^3)

x_s concentration of suspension $(kg\ m^{-3})$

α mass specific filtration resistance $(m\ kg^{-1})$

ε porosity

ψ part of area on which filtration takes place

ϱ density of solid $(kg\ m^{-3})$

μ viscosity (Pa s)

REFERENCES

1. Nývlt J.: *Industrial Crystallization*. Verlag Chemie, New York 1978.
2. Belov V. N., Prutkovskii A. S., Shlyapintokh L. P.: Trudy Len. NII Giprokhim. *15*, 8 (1975).
3. Purchas D. B.: *Industrial Filtration of Liquids*, p. 434. Academic Press, London 1971.
4. Söhnel O., Mareček J.: Krist. Technik *13*, 253 (1978).
5. Söhnel O., Matějčková E.: Chem. Prům. *31*, 285 (1981).
6. *International Critical Tables*, Vol. 5, p. 15. McGraw-Hill Book Comp., New York 1933.
7. Vrána M., Söhnel O.: Czech. Appl. PV-7433-79.
8. Söhnel O., Vrána M.: Chem. Prům. *31*, 242 (1981).
9. Denisova T. I., Kislinskaya G. E., Tkachenko T. N., Kozachek N. N.: Zh. Prikl. Khim. *51*, 523 (1978).